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REPORT OF THE CHIEF OF THE BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY, AGRICULTURAL RESEARCH ADMINISTRATION, 1943

United States Department of Agriculture,
Washington, D. C., October 15, 1943.

Dr. E. C. Auchter,
Administrator of Agricultural Research.

Dear Dr. Auchter: I present herewith the report of the Bureau of Agricultural and Industrial Chemistry for the fiscal year ended June 30, 1943.

Sincerely, W. W. Skinner, *Chief.*

CHEMISTRY CONTRIBUTES TO INDUSTRIAL UTILIZATION OF FARM PRODUCTS

The Bureau of Agricultural and Industrial Chemistry conducts research in a scientific field that has always been recognized as intimately connected with the purposes of the Department of Agriculture. The act of Congress creating the Department in 1862 authorized investigations relating to the application of chemistry to agriculture, and the first scientist appointed in the Department was a chemist. The work of the early chemists dealt with the composition and industrial utilization of fruits, sugar-containing plants, tannin-containing plants, and cereals; also with the composition of soils, fertilizers, feeding stuffs, and insecticides. Thus it concerned both utilization and production of agricultural materials. The volume of chemical work increased and by the time the Commissioner of Agriculture became the Secretary of Agriculture in 1889, the Division of Chemistry included the chemist and a considerable staff of scientific assistants. As originally conceived and developed, this division was the agency in which all the chemical work of the Government should be performed. The work increased steadily in variety and volume, resulting in the elevation of the division to bureau rank in 1901.

The Bureau of Chemistry not only continued to investigate the chemistry of soils, fertilizers, cattle feeds, insecticides, and the various food products derived from cereals, sugar crops, fruits, vegetables, poultry, and farm animals, but it also made chemical and technological investigations of woods, barks, and pine oleoresin as sources of paper pulp, tanning extracts, rosin, and turpentine; it studied the relative value of various fruits and starchy vegetables as sources of denatured alcohol; it made chemical and physical tests of road-building materials; it examined paints, varnishes, inks, papers, leathers, soaps, naval stores, and other products bought on Government contract; and it investigated the production of intermediates for synthetic dyes. However, as other research units of the Department developed

and as new scientific agencies were created in other Government departments, they needed chemists to work in close collaboration with other scientists. Gradually the chemical work that had been initiated by the Bureau of Chemistry on soils, fertilizers, road materials, forest products, dairy products, and meat products was taken over by Federal agencies that were specifically authorized to investigate these materials.

INCREASING ATTENTION TO FOODS

Beginning in the Division of Chemistry, ever-increasing attention was given to foods as the most important products of agriculture. The accumulated evidence that much of the processed food offered for sale to the public was misrepresented as to quality and quantity, and that some was contaminated or deliberately adulterated, led to the passage of the Federal Food and Drugs Act in 1906. The Department of Agriculture was charged with the enforcement of this act, and the Bureau of Chemistry was designated as the agency through which evidence for prosecutions should be obtained by the collection and examination of samples. Similar work was assigned to the Bureau in connection with the enforcement of the Insecticide Act, the Tea Inspection Act, and the Naval Stores Act. So many chemists were required for analyzing samples, for the development of analytical methods, and for research bearing indirectly on the enforcement of these acts, that the regulatory activities seriously interfered with the progress of research in chemistry as related to agriculture. This condition was remedied in 1926 by a reorganization in the Department by which the regulatory work was set up as a separate unit and the agricultural chemical research was combined with the research on soils and fertilizers in the former Bureau of Soils to create the Bureau of Chemistry and Soils.

In later reorganizations the chemical research on insecticides and fungicides was transferred to the Bureau of Entomology and Plant Quarantine, because it related intimately to the work of that Bureau, and the research on soils and fertilizers went to the Bureau of Plant Industry because plant production is wholly dependent upon soils and fertilizers. The remaining chemical and technological research of the former Bureau of Chemistry and Soils was combined with certain engineering research activities of the former Bureau of Agricultural Engineering to create the Bureau of Agricultural Chemistry and Engineering. While in some instances the chemists and agricultural engineers worked on different phases of the same problem, especially when the economy of proposed chemical utilization depended upon low-cost mechanized production of a crop, the engineering research was principally concerned with production, storage, or mechanical processing of a few agricultural commodities, and the chemical research with the chemical processing of a great variety of such commodities and their derived products.

After the establishment of the Agricultural Research Administration to coordinate and direct the various types of scientific research in the Department, the agricultural engineering research was grouped with the research on soils and plant production, and the research on proteins and nutrition was grouped with the research on human nutrition and home economics. The four units of the former Bureau

of Agricultural Chemistry and Engineering remaining in Washington, D. C., and the four regional research laboratories at Peoria, Ill., New Orleans, La., Wyndmoor, Pa., and Albany, Calif., were combined to form the Bureau of Agricultural and Industrial Chemistry.

WORK NOW ON UTILIZATION PROBLEMS

From the time the Department of Agriculture was organized till near the close of the fiscal year 1943 the work of the Chemical Division and its various successors had been concerned in one way or another with production, as well as with utilization, of agricultural commodities. Now it is concerned with utilization alone. However, the chemical and technological problems relating to utilization of agricultural commodities have multiplied rather than diminished with the passage of time, and require concentrated attention from one of the largest chemical-research organizations in the world.

The welfare of this country requires that farmers produce crops best suited to their soil and climate and more than can be consumed by their own families or exchanged with neighbors for other needs. The fourfold larger off-farm population must be fed and clothed, and farmers must profit from sales of produce to insure the production of enough food and fiber to go around. The existence of unsalable surpluses on farms discourages production, and underproduction might lead to disastrous results. Waste of crop residues and portions of crops discarded in sorting to meet market standards or satisfy discriminating buyers increases the prices of goods sold. Agriculture and the Nation as a whole would benefit from the utilization of surpluses, residues, culls, and offgrades of farm commodities. By means of manufacturing processes involving the application of chemical knowledge, fruits and vegetables that are wholesome but unsalable to consumers in the fresh condition because of undersize, oversize, deformity, unattractive color, or other physical defect, might be converted into salable food products. Surplus fruits and vegetables might be preserved in various ways, or manufactured into food products and useful nonfood byproducts, through the application of chemistry and engineering. Surplus grains might be used as sources of starch, dextrin, sugars and sugar sirups, alcohols, glycols, organic acids, and other chemical products. Crop residues might be used in chemical processes to produce cellulose, lignin, and furfural for use in paper, fiberboard, and plastics, and for numerous other purposes.

Even when established industries are using agricultural commodities as sources of nonfood chemical products, chemical research can benefit agriculture by finding new uses for the products, thereby increasing consumption of the raw materials, or by showing how other agricultural commodities might be used for the same purpose. For example: a new large-scale use might be found for furfural, which would increase consumption of crop residues for furfural production; or a plentiful domestic oilseed might be shown to be as useful as a scarce imported oilseed for producing a particular type of oil product through suitable chemical processing of the original oil.

In addition to their application for obtaining diverse food and nonfood products through manufacturing processes, chemistry and related sciences might be applied to decrease the losses of agricultural

commodities and derived products due to microbiological or enzymic action.

The Bureau of Agricultural and Industrial Chemistry is carrying on research designed to benefit agriculture and the Nation as a whole by more complete and more economic utilization of agricultural commodities and residues and their derived products and byproducts; by decreasing deterioration and loss of primary and derived products of agriculture; and by chemical processing of domestic agricultural commodities to meet specific needs for foods and food accessories and for nonfood products including rubber, leather, textiles, paper, fiberboard, adhesives, oils and fats, waxes, resins, plastics, solvents, explosives, medicines, antiseptics, and particular chemical compounds.

The purpose of the regional research laboratories as expressed in the act of Congress authorizing their establishment is—

> to conduct researches into and to develop outlets for farm commodities and products and byproducts thereof. Such research and development shall be devoted primarily to those farm commodities in which there are regular or seasonal surpluses, and their products and byproducts.

In the first and subsequent agricultural appropriation acts providing for the regional research laboratories, the Congress has stipulated that their research should include research on food products of farm commodities. Because of the threat of war, and later the coming of war, the previously planned work of the regional research laboratories, as well as that of other divisions of the Bureau, was realined with more immediate objectives that would contribute to the Nation's war effort. Although agricultural surpluses are practically nonexistent under war conditions, they will undoubtedly require further utilization investigations with the return of peace. The research now in progress will contribute to the original purpose in many ways.

Some of the more outstanding accomplishments of the Bureau during the fiscal year 1943 are related in the following pages. More complete and detailed information concerning some of the Bureau's work is given in the 213 publications issued during the fiscal year, which included 1 Department circular, 1 annual report, 1 article in the Journal of Agricultural Research, 53 processed circulars and information sheets (not published in permanent form), and 157 articles in outside journals and proceedings. Information on newly developed processes and products is given in the specifications of 16 patents granted during the year to employees of the Bureau.

A list of publications issued by the Bureau of Agricultural and Industrial Chemistry, and of patents granted to its employees during the fiscal year 1943, is available in mimeographed form.

FOOD DEHYDRATION PROBLEMS BEING SOLVED

Research intended to facilitate the rapid and intelligent development of a food-dehydration industry adequate for growing military needs and for lend-lease shipments was continued under regular appropriations for the work of the regional research laboratories and for the agricultural chemical investigations of the Bureau. The work on food dehydration in general was done at the Western Regional Research Laboratory. Collateral investigations relating to special applications of dehydration were carried out at the Eastern Regional Research

Laboratory and at the Bureau's field stations in Los Angeles and San Francisco, Calif., Weslaco, Tex., Winter Haven, Fla., and Auburn, Ala.

The engineering work has included studies on the rate of drying of individual fruits and vegetables as affected by each of numerous factors and the preparation of diagrams from the resulting data for use in dehydrator design and operation. Nine new designs for dehydrators and one for a dehydrator tray were prepared and distributed during the year. Two (types K and L) are modifications of previously designed one-truck and two-truck cabinet driers to provide for heating with a coal-burning furnace instead of with steam. One (type N) is a multistage dehydrator, for commercial use, which comprises separate parallel-flow and counter-flow tunnels for two-stage drying; equipment consisting of a group of bin driers may be added for three-stage drying.

Commercial equipment used to prepare vegetables for dehydration was studied with the view of making improvements. Seven drawings representing improved designs for preparation equipment were prepared and distributed. Tests of flame-peeling and brine-peeling equipment were conducted on a pilot-plant scale, and pilot-plant models of a continuous steam blancher and a picking-and-trimming conveyor were constructed and tested. The flame peeler, made as small as was deemed practical, has a capacity of about 50 tons of potatoes per day.

General lay-outs for dehydration plants of various types and sizes were worked out. These include estimates of floor space and plant cost per ton of raw material to be handled in 24 hours, as well as the consumption of water, electric power, and fuel in processing different kinds of vegetables.

During the past year more than 450 separate pilot-plant experiments were made on the dehydration of the more common vegetables and berries in cabinet and tunnel dehydrators, including a commercial cabinet drier, several experimental cabinet driers, and an experimental tunnel drier operated as a countercurrent type or as a multiple-cabinet type. The main purpose of these tests was to determine the most favorable conditions for the dehydration of individual vegetables or fruits to products that are satisfactory with regard to retention of flavor, color, and vitamins during a reasonable period of storage, as well as general appearance, texture, and rapidity of rehydration. Recommendations of dehydration techniques for obtaining acceptable products from most of the common vegetables can now be made, although there still may be possibilities for improvement. Tests showed that potatoes, carrots, and beets dehydrated after cooking could be rehydrated in a very short time.

Numerous pilot-plant experiments have also been made on the spray drying of fruit and vegetable juices and purees and of emulsified whole-egg mixtures. About 150 runs were made with an experimental spray drier and about 25 with commercial spray driers. Powders were obtained which appear to be suitable and practical for the preparation of pureed vegetables for infants and convalescents, and soups, beverages, jellies, and desserts for general use. Many of the dehydrated products compared favorably with the fresh materials with regard to color, flavor, and vitamin content.

In a collateral study of the influence of vitamin intake with the feed of poultry upon the vitamin content of dried whole egg, a definite correlation between vitamin intake and vitamin content of eggs was found in the cases of thiamine and pantothenic acid, and a possible correlation in the cases of riboflavin and vitamin D. There was no appreciable loss of these vitamins, or of nicotinic acid, in spray drying. However, when the dried egg in waxed fiber containers was stored at 98.6° F., 60 percent of the original vitamin A content was lost in 1 month. The loss of vitamin A was much less, and that of thiamine, riboflavin, and pantothenic and nicotinic acids was almost negligible at moderately low temperatures.

Retention of vitamin A by spray-dried whole-egg powder was enhanced to a marked degree by compressing into blocks, and also by adding antioxidants to the emulsified egg mixture before drying. One of the most effective antioxidants investigated was alpha-tocopherol, which may be added as it exists naturally in wheat-germ oil, as derived from vegetable oil, or as produced synthetically. Preliminary experiments indicated that pads of activated charcoal placed inside and at the ends of waxed-paper containers filled with dehydrated whole-egg powder maintained the characteristic odor of the fresh material. These experiments were made under extremely severe storage conditions; namely, in an atmosphere saturated with water vapor and at temperatures of 74° and 98.6° F. for 40 days.

Laboratory tests on the peeling of root vegetables demonstrated that potatoes and rutabagas can be peeled effectively by immersing them in boiling saturated brine for 5 to 10 minutes and then subjecting them to high-pressure sprays of water in a rotary-drum washer. Brine peeling is safer and more economical than lye peeling and is less likely to cause discoloration. The use of brine is advantageous because the boiling brine solution is hotter than boiling water. Peeling losses are substantially lower with brine peeling than with abrasion peeling.

Measurements of the rate of transfer of heat through dehydrated vegetables have shown that these products are very poor conductors of heat. The data have provided a basis for calculating how much time is required for hot-packed dehydrated vegetables to cool to a safe storage temperature. They also have led to a suggestion that dehydrated vegetables might be used successfully as thermal insulation for large-scale shipments of frozen meat in vessels not provided with refrigeration systems, provided practical methods of packaging and stowing could be developed to conserve or supplement the heat-insulating value of the dried products.

Numerous combinations of moisture-resistant sheet materials have been made up into packages and tested for effectiveness in keeping dehydrated vegetables dry and free from insects during shipment and storage. The laminated package containing a layer of lead foil, which is standard under some Government specifications, has been tested and found to be satisfactory with regard to moistureproofness, integrity of seals, and resistance to mechanical shock, changes in atmospheric pressure, and attack of boring insects. Methods for producing a carbon dioxide atmosphere in a sheet-metal can, as required for dried cabbage and carrots, were investigated and improved.

Methods that seem to be commercially practical were developed

for compressing dehydrated vegetables and fruits into compact 2-pound blocks. Such blocks, compared with loose material, represent a saving of almost 50 percent in shipping and storage space, and also a considerable saving in tin plate for packaging. A cubic foot of the compressed food represents from 300 to 500 pounds of trimmed fresh fruit or from 500 to 700 pounds of trimmed fresh vegetables. Tentative specifications were prepared for compressed dehydrated vegetables and fruits.

Difficulties in the dehydration of green lima beans were overcome, and a good dried succotash composed of equal parts of sweet corn and green lima beans was developed for use in Army rations. Bars of mixed dehydrated fruits and nuts were designed for use in the combat ration. Other new dehydrated products include dehydrated lye hominy prepared from both white and yellow corn; tray-dried tomato slices of a particular fleshy variety; a vegetable mixture for use in preparing clam chowder; orange slices that are suitable for preparing marmalade; papaya slices that have a confectionlike texture and flavor; and spray-dried powders, prepared from boysenberries and grapes, that can be converted into excellent jellies by simply adding suitable quantities of sugar and water and cooking for a short time.

At the citrus products laboratory of the Agricultural Chemical Research Division an excellent base for grapefruit marmalade was prepared by dehydrating separately the sliced, cooked peel and a puree made from the rest of the fruit. The pureed pulp is admirably adapted to drying on internally heated revolving drums, even without the addition of sugar.

Experiments were made at the Auburn, Ala., field station by the Agricultural Chemical Research Division for the purpose of developing a suitable farm-scale method of dehydrating sweetpotatoes for use as cattle feed in winter. Data obtained in the dehydration of machine-chipped sweetpotatoes, sweetpotatoes partially dehydrated by pressing after grinding (as in a sugarcane mill), and sweetpotato leaves, using an experimental hot-air tray drier in each case, were used for designing an improved drier, for which a patent application has been filed. These data are also useful guides for operating commercial tray driers to dry sweetpotatoes.

Several engineers and food technologists from the Western Regional Laboratory and two from divisions in Washington acted as instructors in a collaborative dehydration training school held in the fall of 1942, first at the Western Regional Research Laboratory and then in the plant of a food-processing firm in Rochester, N. Y. Courses of lectures and practical demonstrations were given for operators and prospective operators of dehydration plants. These courses were taken by about 250 persons. During 2 weeks in the spring of 1943, conferences on food dehydration, supplemented by lectures and demonstrations, were held at the Western Regional Laboratory for staff members of State agricultural experiment stations.

QUICK FREEZING PRESERVES QUALITY OF FRESH FOODS

The quick-freezing and freezing-storage method of preserving fruits and vegetables, which was unknown less than a generation ago, has made tremendous strides in recent years because this method,

more than any other, preserves the quality of fresh foods and because great progress in refrigeration engineering has made possible the widespread application of this method.

To meet the needs of the food industry, which is hampered by wartime restrictions on the use of metal containers, the Western Regional Laboratory developed techniques for freezing pork and beans, cranberry sauce, tomato products, and the kinds of fruits that were formerly preserved in large (No. 10) cans for commercial pie baking and preserve and jam manufacture.

Wholesome frozen food products of good quality were developed, as a result of war demands, from raw materials usually removed in sorting or preparation for market to increase salability, such as vegetable trimmings and fruits that are distorted, scarred, or overripe. These new products included chopped broccoli, purees of various vegetables and fruits, and steamed-and-boned poultry of stewing age. They are nutritious, palatable, compact, and convenient, and provide an expanded outlet for some good food materials that previously were largely wasted.

Freezing preservation for a limited time has been shown to be practical for prolonging the commercial fruit canning and preserving season. A processing technique developed at the Western Regional Laboratory, which eliminates the necessity of any prefreezing treatment for apricots, prunes, peaches, and pears, will permit packers to can larger quantities of such fruits in spite of factory labor and equipment limitations at harvesttime, if freezing storage is available. Fully ripe fruit was frozen and stored for several months at −10° F. By bringing the temperature to 20°, the fruit is easily pitted or cored and peeled. By finishing preparation of the fruit and placing it in a hot steam kettle for preserves or submerging it in sugar sirup for canning before it was defrosted, discoloration and loss of flavor were prevented.

It was found that strawberries can be frozen with considerably less sugar than usual, without appreciable loss of quality. High-dextrose corn sugar was a satisfactory substitute for cane sugar for this purpose. In tests of strawberry purees frozen in ice cream, addition to the puree of 30 to 50 percent of sugar and 0.5 to 1.0 percent of pectin gave a product that was satisfactory as to color, texture, flavor, and melting properties. The combination of pectin-containing frozen fruit purees with ice cream provides a means for increasing the gallonage of ice cream at a time when there is insufficient cream and milk to meet the demand for ice cream and sherbets. The addition of fruit also provides a greater variety of flavors in ice cream.

A satisfactory formula was developed for a new all-fruit frozen dessert to take the place of ice cream. Officials of the United States Quartermaster Corps have expressed a desire to supply Army camps with this dessert, which has the texture of ice cream and the flavor of fully ripe fruit, and requires no dairy products for its preparation.

Investigations made for the Quartermaster Corps showed that frozen vegetables can be used in Army camps without the installation of extensive special refrigeration equipment, and that, on the basis of semiweekly distributions from refrigerated warehouses to company kitchen refrigerators, spoilage will not occur. In kitchen refrigerators kept at 40° F. spinach (in the case) having an initial temperature

of 20° remained in good condition for 16 days, whereas peas similarly stored showed no deterioration until the eleventh day.

At the request of the Office of Quartermaster General, the Western Regional Laboratory assisted in planning, and advised with regard to the technical aspects of, a program for distribution and use of frozen foods at Army posts in the United States. Under this program 75 million pounds of frozen vegetables would be purchased in the fiscal year 1943–44 for use by the Army. The speed with which this program was adopted and the success of its operation, from the technical standpoint, were made possible by the previous experience and research findings of technical employees of the Bureau of Agricultural and Industrial Chemistry who have been conducting research in this branch of food preservation for the last 10 years. They supply convincing evidence that the research results were useful and that the conclusions drawn from these results were sound.

SALTING AND BRINING PRESERVE VEGETABLES USUALLY CANNED

For several years the Agricultural Chemical Research Division has been cooperating with the North Carolina Agricultural Experiment Station and with pickle manufacturers in chemical, bacteriological, and technological studies to solve the problems connected with commercial production of good cucumber pickles, which in the past was difficult under southern climatic conditions. Recently this investigation has been broadened to include studies on the preservation of other vegetables by salting, brining, and acidic fermentation as wartime emergency methods for preserving vegetables that otherwise might be wasted due to a lack of suitable containers or equipment for canning, or of facilities for dehydration or freezing preservation.

Four general methods have been tested for the salt preservation of lima beans, snap beans, beets, cabbage, carrots, cauliflower, corn, greens of various kinds, lettuce, okra, onions, peas, peppers, rutabagas, green tomatoes, and turnips. These are: (1) Dry salting with a small amount of salt; (2) dry salting with a large amount of salt; (3) brining with weak brine plus vinegar; and (4) brining with strong brine. On the basis of the experimental results, the relative values of the different methods for particular vegetables have been determined. In most cases one method will give the best results for a particular vegetable, but for some vegetables any one of two or three methods will give satisfactory results. In the light-salt and weak-brine methods the preserving effect is due principally to lactic acid formed by bacterial fermentation of sugars drawn from the vegetable material by the salt or brine. In the heavy-salt and strong-brine methods, the salt is the principal preservative.

These salt preservation methods are advantageous because they are simple and inexpensive; they require no heat for some vegetables and comparatively little for others; processing is done in wooden, earthenware, or glass containers instead of in metal; and the products can be kept in large or small quantities in closed, impervious nonmetal containers without hermetic sealing, provided the vegetables are entirely covered by brine.

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RESEARCH ON ALLERGENS DISCLOSES RELATIONSHIP BETWEEN CHEMICAL COMPOSITION AND ALLERGENIC ACTIVITY

Further research on the allergens of agricultural products was carried on during the past fiscal year under a Bankhead-Jones basic-research project supported from the special research fund.

Information from research on allergens of cottonseed, assembled in previous years, acquires new importance in wartime. Restrictions in the supplies of meat and other animal proteins have directed attention to plants as sources of acceptable proteins for the human dietary. Supplies of important animal fats are likewise inadequate to meet current needs for both food and munitions. Chief sources of vegetable proteins suitable for replacement of animal protein are the oilseeds, represented principally by cottonseed, peanut, and soybean. These seeds are also sources of edible fats. Various nuts comprise an additional source of excellent food protein and fat. Castor bean production and milling must be expanded to furnish oil for special military use.

Among numerous allergens encountered in agricultural products those of the oilseeds and nuts are exceptionally potent. Evidence secured from study of allergens of cottonseed identified the major allergenic component as a protein of the type called natural proteose. Prior to their recognition as allergens by this Bureau the proteoses had received little study. Proteose fractions were separated from currently important edible oilseeds and from castor bean for determination of their chemical composition and physiological properties. Results indicate a close relationship between proportion of proteose and allergenic activity. A proteose from castor bean was the most potent allergen yet encountered among the oilseeds. Minute quantities of this allergen are capable of causing disabling allergic disorders. This evidence points out the source of industrial hazards in production and utilization of castor beans. Additional study of this component is under way to determine its significance in utilization of castor bean residues from recovery of the oil.

Closely associated with the allergenic proteoses of both cottonseed and castor bean is complex carbohydrate. Experiments have shown that this carbohydrate does not determine the specific allergenic activity of the proteose but does influence the degree of activity, particularly the capacity of the proteose to induce a sensitiveness manifested by allergy.

Recent experiments dealing with the stability of allergens afforded data on the mode of action of these natural protein components. Chemical linkages between the amino acids that make up the allergen were not disrupted by boiling with dilute acid, and the property of inducing characteristic skin reactions was not altered by this treatment. The fact that this drastic chemical treatment virtually destroyed the original capacity of the allergen to induce sensitiveness and to produce other typical allergic reactions signified that structural rearrangements had occurred. Apparently, however, the capacity of an allergen to induce reactions when applied to skin depends on different qualities from those required to induce sensitiveness and to enter into reactions from which usual signs of allergy are generated.

Further indication of the extraordinary complexity of the allergic response to proteins was the demonstration that a second allergen exists among the proteins of cottonseed. This one is a globulin, or is intimately associated with a protein of this type. Some allergic individuals are sensitive only to this recently discovered allergen; some are sensitive only to the one previously identified; others respond to contact with either allergen. This evidence clarifies apparent variations in response induced in different individuals by the same product.

Certain allergens of incubated eggs were investigated during the past year in relation to a greatly expanded production of vaccines from this source, brought about by the war. This study was the basis of identification of allergenic egg protein in vaccines and showed that allergen present in the vaccine was derived from the egg and not from the inoculated organism. Presence of egg allergen in a preparation intended for injection represents a potential hazard owing to the possibility of either sensitizing the recipient to egg in foods, or causing severe allergic reaction if the recipient is inherently sensitive to egg protein.

PRODUCTION OF PENICILLIN ACCELERATED BY RECENT RESEARCH

By greatly increasing yields of penicillin, a remarkable mold-produced drug which inhibits the growths of streptococcus, staphylococcus, and other pus-forming germs, the Northern Regional Research Laboratory has helped to speed the day when this boon to mankind can be produced commercially at nonprohibitive cost and in sufficient quantity for use in civilian, as well as Army and Navy, hospitals.

Penicillin was discovered in 1929 by a British bacteriologist, Prof. Alexander Fleming, who observed a germ-free zone surrounding a small particle of mold which contaminated an agar-plate culture of a staphylococcus species. The mold was identified as *Penicillium notatum* and consequently the germ-inhibiting substance excreted by the mold was named "penicillin." Further investigation was stimulated by the war. In 1940 other British investigators, led by Prof. H. W. Florey and Dr. E. Chain of the University of Oxford, succeeded in preparing a small quantity of the drug in concentrated form, which was administered to human patients for the first time in 1941, with marked success.

Professor Florey was so impressed with the possibilities of this drug for treating gangrenous wounds and infected burns that he and an associate, Dr. N. G. Heatley, came to the United States to urge that attempts be made to produce the drug commercially. He was encouraged by the Chemotherapy Committee of the National Research Council and the Committee on Medical Research of the Office of Scientific Research and Development of the Office for Emergency Management, which arranged to support concentrated research for a limited time on penicillin production at the Northern Regional Research Laboratory by its Division of Fermentation Research, in cooperation with Dr. Heatley and four other scientists, representing the Office of Scientific Research and Development.

After a preliminary survey of possible penicillin-producing organisms, a careful comparison under identical conditions was made of the penicillin-producing ability of 4 cultures of *Penicillium notatum* obtained from different research organizations, but all derived from Fleming's original culture, and 36 assorted, but closely related,

penicillia from the large culture collection of the Northern Regional Research Laboratory. It was found that 5 molds out of the 36 from the laboratory's collection had the ability to produce penicillin to the same extent as the 4 strains of Fleming's organism. These 9 best penicillin producers were selected for experiments on increasing yields.

In preparation for these experiments, comparative studies were made of conditions that had been used by various research organizations and individuals to promote the production of penicillin, on the various procedures for preparing penicillin concentrates, on the potency of penicillin preparations, and on bio-assay methods for making quantitative determinations of penicillin in terms of Oxford units per milligram.

The study of factors involved in the various methods of bio-assay has led to a refinement of these methods and greater accuracy of results. Two methods are available, an agar-plate method, which is better suited to routine work, and a broth-dilution method, which is believed to be more accurate.

Use of lactose or starch, instead of glucose, as the source of carbohydrate in the medium for growing the mold, together with minor changes in the medium, increased the yield of penicillin substantially.

New strains of *Penicillium notatum* were recently found which are especially well adapted to penicillin production in either surface or submerged fermentations. By using these better strains in combination with the better medium, yields up to 220 Oxford units per milliliter were obtained. These yields were about 100 times as great as those obtained when the work started.

As a result of intensive study on the purification of penicillin, concentrates were obtained, by means of chromatographic adsorption, which had a potency of 2,000 Oxford units per milligram, whereas a year previously, when the work was begun, the highest potency reported from any research laboratory was 40 units per milligram. This concentrate was not yet pure, but it was believed that the attainment of maximum potency or even crystalline penicillin was not far distant. (Since the end of the year, this has been achieved.)

From experiments on the recovery of penicillin from fermentation liquors it appears that a product sufficiently pure for clinical use (100 to 250 Oxford units per milligram) can be obtained by simple methods and equipment, and that chromatographic adsorption will not be required for this purpose.

With the improved fermentation processes developed by the Northern Laboratory as a basis, the commercial production of penicillin has been undertaken by 17 chemical and pharmaceutical manufacturers, and the output of this powerful new antiseptic has been increased from a few million units to approximately 7 billion units per month. Thus far, practically all of the product has been requisitioned by the Government for use by the armed forces.

Because of its own contributions and leadership in cooperative research on the development of improved fermentation processes, assay techniques, and procedures for isolating and purifying the drug, the Northern Laboratory has become the center for consultation and instruction on the manufacture of penicillin in this country.

SYNTHETIC RUBBER OBTAINABLE FROM GRAIN VIA BUTYLENE GLYCOL AND BUTADIENE

When the outbreak of war with Japan threatened to cut off importations of natural rubber from the East Indies, the Northern Regional Research Laboratory, having a staff of chemists and microbiologists experienced in fermentation research, concentrated such research on the production, from agricultural materials, of chemicals useful for making synthetic rubber. Butylene glycol, which has received research attention at Iowa State College in recent years, as a possible commercial fermentation product of corn, seemed to be a promising intermediate for the production of butadiene, which could be polymerized to synthetic rubber by newly developed commercial methods; so the problems that remained to be solved before commercialization of butadiene production from carbohydrates by way of butylene glycol were attacked with all available personnel and facilities. Three steps are involved in the scheme; namely, fermentation of carbohydrate to 2,3-butylene glycol, recovery of pure butylene glycol from the beer (fermentation liquor), and conversion of the butylene glycol to butadiene. Before July 1, 1942, a great many laboratory experiments had been carried out, and new knowledge had been gained with regard to the first and second steps; also, research had been started on the third step. During the fiscal year 1943 pilot-plant experiments were made in all three steps, supplemented by necessary laboratory control and research work.

The first fermentation experiments had been made on glucose or corn-sugar solutions. When starch or flour mashes were fermented, after addition of malted barley to convert the starch to sugars, the yields were lower, obviously because of incomplete hydrolysis. In attempts to remedy this difficulty, experiments were made on the use of malt and moldy-bran extracts and on the symbiotic cultivation of the glycol-producing organism, *Aerobacter aerogenes*, with diastase-producing fungi. Occasionally higher glycol yields were obtained, but these higher yields could not be reproduced at will, and it was concluded that controlled symbiotic cultivation of glycol-producing and diastase-producing organisms involves such serious operating difficulties as to be impractical at this time.

Experiments were also made on the fermentation of mashes, made of starch, granular wheat flour, or corn grits, which had been hydrolyzed with acid and then neutralized. Fermentation proceeded more rapidly than with glucose solutions, but the yield of ethyl alcohol, which is a minor product of the glucose fermentation, equaled or exceeded that of butylene glycol.

Isolation of a new bacterial organism permitted the direct fermentation of ground grain to the levo-rotatory isomer of 2,3-butylene glycol and ethyl alcohol, in the ratio of about 2 to 1, because this organism itself produces sufficient diastase to convert the starch to fermentable sugar. Although the total quantity of products was greater than that obtained with the other organism, the fermentation was slower and the yield of butylene glycol was lower per unit quantity of grain processed. Therefore, the process was not considered to be practical for the purpose in mind. The levo-2,3-butylene glycol, an entirely new chemical compound, differs from the previously obtained isomer, meso-2,3-butylene glycol, in other properties as well as in optical rota-

tion. For example, the former can be used in automobile radiators as an antifreeze, like glycerine, whereas the latter cannot be used for this purpose because it forms a high-melting hydrate.

Recovery of glycol from grain mashes was found to be more difficult than from fermented glucose solutions, and less complete, because the high proportion of suspended solids in the former interferes with filtration and evaporation. It is believed, however, that a recently devised recovery system will overcome the difficulties. It includes partial evaporation of the strained beer in multiple-effect evaporators, and final evaporation of the thick, sirupy evaporator residues, which retain considerable glycol, on metal drums heated internally with steam under pressure; also, separate condensation of the vapors from the two types of evaporators, and combination of the condensates for rectification. This proposed recovery method, which would require only equipment and operations similar to those employed in conventional grain distillery practice, where distillation residues are recovered by means of drum drying, remains to be tried out on a large scale. A 92-percent recovery of butylene glycol from fermentation liquors has been obtained in small-scale experiments, and better than 95-percent recovery is believed to be possible.

Conversion of butylene glycol to butadiene by first forming the diacetate of butylene glycol and then cracking this derivative at high temperature was studied in a pilot plant, designed and built at the Northern Regional Research Laboratory, which is capable of producing 100 pounds of butadiene per day. This work was completed and a full report submitted to the Office of the Rubber Director. The results showed that more than 85 percent of the butylene glycol can be converted into butadiene having a purity of 98.5–99 percent, and that 99 percent of the acetic acid used in the process can be recovered. Small amounts of salable byproducts can also be recovered. In the conversion of butylene glycol to its diacetate, yields equivalent to 97 percent of the theoretical yield were obtained.

The report to the Office of the Rubber Director described the experimental process and equipment and gave operating data necessary for the design of a large-scale plant. The conversion of butylene glycol to butadiene requires special equipment, whereas the fermentation process and the process of recovering glycol from the fermentation liquors can be carried out with equipment of standard design, well known in the grain-distilling industry. The information available at the close of the fiscal year was believed to be sufficient for designing a full-scale plant for obtaining butadiene from grain since all the engineering details, except the exact capacities of certain equipment used in the fermentation and recovery steps, are available to design engineers.

On the basis of 12.5 pounds of butylene glycol as the fermentation yield per bushel of grain, and a 92-percent recovery of glycol from the beer, both believed to be conservative figures, a bushel of corn or wheat will produce 5.9 pounds of butadiene. By increasing the yield of glycol to 13 pounds per bushel and the recovery of glycol to 95 percent, both of which seem possible, a bushel of corn or wheat would yield 6.3 pounds of butadiene. It is understood that the alcohol-to-butadiene process now in operation yields from 5.25 to 5.5 pounds of butadiene per bushel of corn.

RECOVERY OF RUBBER FROM DOMESTIC PLANTS UNDER INVESTIGATION

During the past fiscal year the Bureau of Agricultural and Industrial Chemistry participated in the Emergency Rubber Project of the Department of Agriculture, which was under the leadership of the Forest Service. The Eastern Regional Research Laboratory investigated chemical and engineering problems relating to the extraction and processing of rubber from guayule, kok-saghyz, cryptostegia, rabbitbrush, and pingue, while the Southern Regional Laboratory gave attention to similar problems in connection with goldenrod.

Commercial methods for recovering rubber from wild guayule presented difficulties when applied to the immature 2-year-old cultivated shrub which forms the basis of the present program. Pilot-laboratory studies resulted in an improved process which overcomes the obstacles and may enable factories to operate 12 months of the year instead of 6 as at present. In the application of the modified process, the harvested shrub will receive a quick partial drying followed by removal of leaves and flower stems by tumbling in a simple revolving drum. Part of the defoliated product will then be further dried for immediate processing, while the remainder will be baled for storage. It will be taken from storage as required, cut to ⅛-inch or 1/16-inch mesh on a high-speed rotary knife cutter, and sent directly to pebble mills. The resulting worms of coagulated rubber will be floated free of the comminuted plant fiber, subjected to a clean-up process, and dried. The bagasse will be dewatered by pressure, and, together with the leaves, used as fuel.

Defoliation, applied to young shrubs harvested at the proper season, reduces by 25 to 35 percent the weight of material to be processed and actually increases by 5 percent the amount of rubber obtained. It also reduces the water-to-solids ratio necessary for pebble milling, which further increases the capacity of the mills, in some cases by as much as 100 percent. The purity of the recovered rubber is also higher. It contains about 65 percent of pure rubber hydrocarbon and has a tensile strength about half as great as that of hevea rubber.

A reduction in the cost and amount of equipment required per ton of shrub was achieved through the substitution of light, relatively inexpensive rotary knife cutters for the customary train of three pairs of massive crushing rolls.

It was established that retting young guayule under easily controllable conditions will improve the quality of the rubber extracted by pebble milling. In one case the rubber produced after 4 days of fermentation contained 69 percent of pure rubber hydrocarbon as compared with 60 percent before retting, and the tensile strength was increased to 60 percent of that of hevea. Much work was done to adapt milling procedures to this retted material.

Equipment was designed and ordered for a large-scale integrated pilot plant at Salinas, Calif., for demonstration of the commercial practicability of the pilot-laboratory results.

About 10,000 pounds of fresh kok-saghyz roots were processed to recover rubber by various means. A method was developed for isolating 50 to 60 percent of the total rubber in the form of a concentrated latex. Also a commercially practicable method was developed on a small-batch pilot-plant scale for efficient recovery of the rubber from

kok-saghyz in the form of solid worms by a series of short pebble millings with intermediate screening operations. The quality of the product is good, being intermediate between that of guayule and hevea rubbers. The resin content is about 12 percent and the content of benzol-insoluble matter, 5 percent.

From these preliminary experiments, it appears that kok-saghyz would constitute a desirable source of good-quality rubber if a sufficient supply of the roots were available. Fermentable carbohydrate would be a valuable byproduct. Equipment was designed and ordered for the construction of an integrated large-scale pilot plant at the Eastern Regional Laboratory for recovering kok-saghyz rubber by the mechanical process.

Exhaustive tests of methods for obtaining rubber from harvested *Cryptostegia grandiflora* showed that recovery by any of the techniques yet devised is not commercially feasible. Furthermore, the quality of the rubber obtained from harvested plants was distinctly inferior to that obtainable from the latex drawn from living plants.

Work at the Eastern Regional Laboratory has demonstrated that rabbitbrush (*Chrysothamnus nauseosus*) is not promising as a source of rubber.

Recovery of rubber from pingue, or Colorado rubberweed (*Actinea richardsoni*), was demonstrated as practical by the conventional guayule pebble milling process. If pingue is available in locations permitting its harvest in reasonable quantities, it can be processed in guayule factories to yield a rubber as good as guayule rubber except for its high content of copper and manganese.

Work on a laboratory scale established the possibility of recovering most of the rubber in goldenrod plants by extracting the dried and pulverized leaves with benzol after a preliminary extraction with acetone to remove resinous material. It is believed that this process could be carried out commercially in wood-rosin extraction plants. A single-unit extractor was used for the 1942 experimental crop of goldenrod to obtain engineering data on the principal operations and to produce goldenrod rubber for laboratory testing. A larger eight-unit extraction plant was built to handle the larger supply of goldenrod from the 1943 crop. It provides sufficient capacity for extracting 40 to 50 pounds of rubber from 1,000 pounds of dry leaf material containing 6 percent of rubber per day in a semicontinuous cycle.

Two usable forms of goldenrod rubber were produced. One is uncompounded raw rubber that can be blended with Buna-type synthetic rubber. The other is prepared by adding a portion of the compounding agent to the benzol solution and partially curing. After removal of the solvent this can be milled and completely compounded as a workable tire-tread stock containing goldenrod rubber alone. Adequate quantities of both products were produced for processing, vulcanization, and physical testing, and their properties were studied in collaboration with three rubber companies.

When goldenrod rubber was blended with Buna-S synthetic rubber in ratios ranging from 10:90 to 50:50, the resulting stocks compared with Buna-S alone had increased building tack and elongation, and reduced hysteresis and heat accumulation. The goldenrod rubber alone compounded in benzol solution was milled, extruded or calendered, and vulcanized to products having high tensile strength and other desirable physical properties.

NOREPOL, A RUBBER SUBSTITUTE, MADE AVAILABLE TO INDUSTRIES

The process for making a rubberlike material, consisting of polyesters of the fat acids from soybean and other oils high in linoleic acid, was discovered by the Northern Regional Research Laboratory and disclosed to industry about the end of the previous fiscal year. Within 6 months the new polymer, named Norepol (NOrthern REgional POLymer), was being produced on a commercial scale. Several firms used essentially the same process as that developed by the Northern Regional Laboratory but marketed their products under proprietary trade names. The unusually rapid transfer from pilot plant to commercial production resulted from wholehearted cooperation of some chemical firms engaged in the processing of seed oils. The evaluation of Norepol as a substitute for rubber in various applications, and as a new material for mechanical products, was also accomplished within a short time by a large number of manufacturers.

Norepol is not suitable for making automobile tires or for use as tire retreads in its present stage of development. However, it has been used to replace rubber in a number of applications, including tubing, arch supports, jar rings, plumbing gaskets of various shapes and sizes, waterproof fabric, and adhesives. Since jar rings are subjected to very severe conditions in pressure cookers, which cause Norepol to become plastic with loss of tensile strength and elasticity, it is necessary to incorporate a small amount of reclaimed rubber with a Norepol mixture intended for jar rings.

Norepol is resistant to water, dilute mineral acids, lubricating oils, some organic solvents, and severe weather conditions, but is damaged by alkalies, organic acids, and most of the common volatile organic solvents. Two important properties that enhance its usefulness for certain purposes are its resistance to rapid oxidation by ozone and its flexibility at extremely low temperatures. It remains flexible after exposure to ozone for 7 hours. It also remains flexible for long periods at $-20°$ F. and for short periods at $-40°$.

The relative position of polyester elastomers from seed oils (Norepol and similar commercial products) among industrial materials for wartime uses will depend on the demand for rubber substitutes, on the availability and price of suitable seed oils, and on whether or not metals for manufacturing equipment can be spared from other demands of the war effort.

Research on the fundamental chemistry of the reactions involved is being continued at the Northern Regional Laboratory. Marked improvements in the physical and chemical characteristics of Norepol have been achieved, using laboratory equipment, but commercial exploitation awaits further research and development.

NORELAC, A NEW RESIN, MADE FROM VEGETABLE OILS

As a corollary to its work on the development of the rubber substitute, Norepol, the Northern Regional Research Laboratory has investigated a large number of derivatives of polymeric fat acids obtained from vegetable oils. One of the more interesting of these derivatives is the polyamide of residual dimeric fat acids, which has been named "Norelac." The preparation of this substance differs from that of Norepol principally in that the dimeric acids or their esters are condensed with ethylene diamine, instead of ethylene glycol.

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Norelac is a thermoplastic resin which shows excellent adhesion on most surfaces; it has good resistance to alkalies, acids, vegetable oils, and some organic solvents; and it is resistant to the passage of water vapor. These properties suggest its use as a heat-sealing, laminating, and moistureproofing agent for the preparation of containers and wrappers for food products, chemicals, precision machine parts, etc. Norelac appears suitable also for a shellac substitute and lacquer constituent; but at present such uses do not appear sufficiently important to warrant allocations from critical vegetable oil supplies.

Preparation of Norelac on a small pilot-plant scale indicated that little difficulty should be encountered in large-scale production, at a basic manufacturing cost of 32 to 35 cents a pound. Details of the preparation and properties of Norelac were disclosed to potential manufacturers and users of the product at an industrial conference held at the Northern Regional Laboratory.

NORESEAL, A SUBSTITUTE FOR CORK IN BOTTLE CAPS, MADE FROM FARM PRODUCTS

Very satisfactory progress has been made by the Northern Regional Research Laboratory in the development of a cork substitute in the form of thin disks for lining crown-seal bottle caps. The new product, which has been named "Noreseal," is composed of about 30 percent of peanut shell pith together with proteins and plasticizers, all obtained from agricultural products.

Because of its flavor and the necessity for pasteurization, beer is by far the most difficult beverage to bottle satisfactorily with caps having a cork substitute lining. However, practical bottling tests by 5 large breweries on more than 6,000 bottles of beer have demonstrated that Noreseal is equivalent to cork under even the most severe conditions of handling and storage. Tests by other bottlers have shown that the disks perform satisfactorily with soft drinks as well.

Preliminary manufacturing details, based on a small pilot plant, have been worked out, and a larger pilot plant is being designed. In this plant, to be financed by an industrial association, problems of full-scale manufacture will be worked out.

While restrictions on the use of cork have been eased somewhat in recent months, there is evidence that Noreseal, because of its desirable properties, will find industrial use.

NOREPLAST, A NEW TYPE OF PLASTIC, USES FARM WASTE

Bottle caps have been molded commercially, and pencil and pin trays, drinking cups, fuse parts, automobile distributor heads, and various other articles have been molded experimentally, from thermosetting plastic mixtures of a new type containing a major proportion of crop byproducts such as wheat straw, cornstalks, peanut shells, and flax shives. This new molding compound, developed by the Northern Regional Research Laboratory and named "Noreplast," not only provides a profitable use for the ligno-cellulose residues from field crops, but also substantially reduces the proportion of critical phenol-formaldehyde resin required in thermosetting molding compounds.

In current commercial practice, the basic mixture for molding articles that should not lose shape on reheating contains phenol-

formaldehyde resin and wood flour in about equal parts by weight. The new product, however, contains only 25 percent of phenol-formaldehyde resin, as well as 15 percent of special resinous plasticizer and 60 percent of agricultural residue.

Large-scale tests made by a manufacturer on bottle caps molded from the new type of plastic showed that they have sufficiently high torque strength, low water absorption, and high resistance to 50-percent alcohol, to meet commercial requirements. In addition, the caps would be readily molded at the high speed required in commercial operations.

WHEAT BECOMES IMPORTANT TO CHEMICAL INDUSTRIES

Increased demands for industrial alcohol, accompanied by shortages in supplies of corn and restricted importation of molasses, made it imperative that wheat be used as a raw material for the production of alcohol. Because of foaming in the still and other operating difficulties, many distillers were reluctant to change from corn to wheat. The problems involved in the use of wheat for alcohol manufacture were discussed with representatives of the industry in conferences held at the Northern Regional Research Laboratory. Through cooperative research conducted in the Bureau's laboratories and at various distilleries, on request of the War Production Board, the main operating difficulties were overcome and wheat was accepted by the industry as a suitable raw material for the production of alcohol. Wheat used for alcohol production during the fiscal year 1943 amounted to about 54,000,000 bushels.

A process developed by the Enzyme Research Laboratory is expected to contribute materially to the utilization of wheat for alcohol production and to permit wheat to compete with corn whenever the price differential is satisfactory. Wheat contains the enzyme diastase (the active principle of malt) in sufficient quantity to convert all of its starch into fermentable sugar, but this diastase is inactive in the grain and its milled products, because it is combined with protein to form an insoluble complex. The new process makes wheat diastase available, thus permitting a reduction in the proportion of malt used, if not entirely eliminating the need for malt. It is based upon the facts that sulfite solutions activate the proteolytic enzymes of wheat and that the activated proteolytic enzymes partially digest the proteins, liberating the diastase in soluble form.

In this process, which has been tried out on a pilot-plant scale with encouraging results, ground wheat or wheat flour is mixed with very dilute sodium sulfite solution in a tank. Suitable mechanical treatment causes clotting and flotation of the wheat proteins, which are skimmed from the surface. If flour is used, the protein is fairly pure and suitable for use as food; if ground whole wheat is used, the protein is mixed with bran, but the mixture is suitable for feed. The starch settles to the bottom of the tank. The supernatant clear solution of diastase is decanted into a second tank and the starch remaining in the first tank as a sediment is gelatinized by cooking with steam. After the starch paste cools, the diastase solution is returned to the first tank and mixed with the starch. After conversion of the starch to sugars, yeast is added and the fermentation proceeds in the usual manner. This process is adaptable to existing alcohol plants without much change in plant equipment. It may require

somewhat more time than the usual grain-fermentation process, using malt alone for saccharification, and the alcohol may contain sulfur which would require removal to comply with existing specifications. Means are being sought for overcoming these objections. Satisfactory yields have been obtained in the pilot-plant experiments.

Two new processes for the recovery of starch from wheat were developed at the Northern Regional Laboratory. One of these is applicable to whole wheat and resembles the process used in wet-milling of corn in that it involves steeping in dilute sulfurous acid solution. Experiments on a pilot-plant scale showed that this process gives about the same high yields (around 82 percent) and good quality of starch from damaged (sour or musty) wheat unfit for food as from sound wheat of the same type. Hard wheats were found to be amenable to processing by this method but, as expected, gave smaller yields of starch. In this process the byproduct gluten loses its plastic and adhesive properties but is believed to retain its feeding value. Most of the gluten can be recovered.

The other process is applicable to ordinary and granular wheat flour from both soft and hard wheats. It comprises: Treatment of the flour with dilute aqueous solution of alkali to peptize or dissolve the protein; separation of the starch by tabling, settling, or centrifuging; washing the crude starch to remove adhering protein solution; neutralizing the alkali remaining with the starch; screening the starch to remove fiber; washing the starch until free of salt; and drying. Protein may be recovered from the alkaline solution by precipitation with acid. This process is believed to be practical for the production of (1) high-quality dried starch for ordinary uses, (2) starch slurries for use in fermentation industries, (3) starch suitable for conversion into glucose sirups or sugar, and (4) byproduct gluten of high protein content. Practically all of the starch in the flour can be recovered, about 80 percent as starch of highest quality, and the remainder as starch of sufficient purity for fermentation or conversion to glucose. Around 60 percent of the protein, in sufficient purity for use in producing glutamic acid for condiments, can be recovered. The process has been shown to be applicable also to the flours of rye, barley, and oats, but starch recovery is less than when wheat flour is used.

Because of the critical corn situation, details of these two wheat-starch processes were disclosed at an industrial conference held at the Northern Regional Laboratory in May, and arrangements were made for further intensive study of both methods, on a pilot-plant scale, in the regional laboratory and in the laboratories of several interested industrial firms.

The Western Regional Research Laboratory started an investigation on the preparation of glutamic acid from wheat protein for use in condiments to give a meatlike flavor. A method for the separation of glutamic acid from other amino acids by use of commercial synthetic resins having anion-exchange properties was developed. This may have industrial value. An improved method for the quantitative determination of glutamic acid in proteins was devised and made available for industrial use.

A sulfate derivative of wheat gluten was found in preliminary tests to accelerate the healing of wounds. Contacts have been made with surgeons and pharmacologists to determine more definitely the possi-

bility of using gluten sulfate for this purpose. In the form of a powder, this substance absorbs about 200 times its own weight of water to form a water-insoluble gel.

DOMESTIC STARCHES SHOW POSSIBILITIES FOR NEW INDUSTRIAL USES

Recent research on the production of the synthetic rubber intermediates, butylene glycol and butadiene, and on the manufacture of starch from wheat, is described in preceding sections of this report.

Starch research of the Northern Regional Laboratory was concerned with the production of so-called waxy grain starches from particular varieties of corn and grain sorghum having seeds described as glutinous. Starches were extracted from most of the available varieties of glutinous corn and sorghum by a method closely approximating that used for the commercial production of cornstarch. Sorghum gave processing difficulty in proportion to the amount of color in the seed coat. However, good-quality starch was obtained after removal of the coat by pearling. The starches from glutinous corn and sorghum were found to have properties similar to those of tapioca starch, and to yield dextrins superior to those from tapioca. All available information on the production and characteristics of waxy grain starches was given to representatives of industrial firms in a conference held at the Northern Regional Laboratory in October 1942. It was expected that small commercial crops of glutinous corn and sorghum would be produced in 1943 and that the 1944 crops would meet all industrial demands that might develop by that time.

Many methods proposed for separating starch into two fractions were tried out and two or three seemed to be commercially practical. The fractions differ in physical properties, and the probable explanation is that the molecules of one fraction are linear while those of the other fraction are branched. The fraction having linear molecules, which is the more promising as a raw material for films, fibers, and plastics, constitutes about 25 percent of cornstarch. When acetylated, it can be made into strong, pliable transparent films like Cellophane, whereas the other fraction, when acetylated, yields weak, brittle films. It was found that the starch acetate films made from the former fraction are very similar to cellulose acetate films and can be plasticized with most of the plasticizers used for cellulose actetate. However, less plasticizer is required for starch acetate, 10 to 20 percent being sufficient to impart any desired degree of plasticity. Flow tests made on starch acetate from the fraction having linear molecules showed that it is a good thermoplastic suitable for molding.

Experiments with starch acetates prepared from the two fractions of starch mixed in known proportions showed that mixtures containing up to 50 percent of the fraction composed of branched-chain molecules yielded actetates having good film-forming quality; therefore about half, instead of one-fourth, of cornstarch is useful for film production. An improved method for starch acetylation was developed which converts starch to starch acetate very rapidly without degradation of the starch. Starch acetates were prepared from unfractionated corn, wheat, glutinous corn, potato, and sweetpotato starches and compared with regard to physical properties. In general, these were very similar, but the acetate from glutinous corn-

starch was less viscous, and that from potato starch was less soluble in organic solvents, than the others. In view of the general similarity, it is likely that cornstarch, which is most abundant and cheapest, would be the only one used in large-scale production of starch acetates or analogous derivatives.

Research was also conducted at the Northern Regional Laboratory on the production of needed organic acids, and other chemicals, from starch indirectly or directly. Extensive experiments were made on the production of itaconic acid by mold fermentation of corn sugar, using numerous cultures from mold spores which had been irradiated with ultraviolet light to produce mutations, and with cultures of other molds which had been isolated from soil and selected for their itaconic acid-producing ability. Using the best organism and the best culturing conditions known, it was possible to ferment corn sugar to itaconic acid in yields close to 40 percent of theoretical, or 30 percent on weight of sugar consumed. Itaconic acid is a substituted methacrylic acid and, in combination with the latter, can be used to produce transparent plastics of greatly improved strength. However, its price (about $10 a pound) has been too high for such use. By means of the improved fermentation process 20 cents' worth of corn sugar will yield a pound of itaconic acid, which can be recovered from the fermentation liquor by a simple and commercially practical method. It was found possible to obtain saccharic acid, in yields of 40 percent or more of theoretical, by controlled oxidation of corn sugar with nitric acid. These yields were about twice as high as those reported by other workers. Saccharic acid is useful for producing synthetic fibers, plastics, and plasticizers and as a replacement for tartaric and citric acids, used principally in food products.

Significant progress was made in the development of mild chemical reactions for converting starch and its hydrolysis product, dextrose, into water-insoluble polymers having plastic properties. A transparent, viscous sirup composed of mixed acetates and propionates of dextrose was produced in good yields by reacting dextrose with acetic anhydride and propionic acid in pyridine. This product showed promise of being useful as a plasticizer. By effecting cross-polymerization of starch and dimeric linoleic acid, in conjunction with acetylation, products were obtained which were insoluble in all solvents tested. Insolubility is a necessary characteristic of plastic for use under stringent conditions.

At the Eastern Regional Laboratory a method was developed for preparing highly substituted starch ethers having plastic properties. When such derivatives were compounded on conventional rubber-working rolls with pigments, sulfur, and accelerator and were subsequently vulcanized, a product having a smooth glossy surface and resembling hard rubber in many respects was obtained. The product was insoluble in all common organic solvents; was resistant to oil at 100° C.; and was not attacked by dilute acids or alkalies. It appeared to be useful for surfacing veneer panels, table tops, and counters. A paper dealing with the recovery of starch from potatoes and another with chemical derivatives of starch were prepared for the 1943 fall meeting of the American Chemical Society.

With technical assistance and advice from the Southern Regional Laboratory numerous improvements were made at the Laurel, Miss., sweetpotato-starch plant, operated by a cooperative association of

sweetpotato growers, to increase its output of high-quality root starch for special wartime requirements. Simple and reliable methods for improved control of starch bleaching and other processing operations were developed and applied on a plant scale, with resultant economy of chemicals and improved quality and uniformity of product. Laboratory and pilot-plant tests were made on new high-starch varieties of sweetpotatoes, recently released for commercial production, to learn the best conditions for handling them in the starch factory. Most of the sweetpotato starch produced in 1942 was sold as fast as produced for special uses for which sweetpotato starch has been found to be better than other available starches. Among such uses were filler for dry batteries and coating for electrical insulating cloth.

LACTIC ACID IS SOURCE OF RESINOUS AND RUBBERLIKE PLASTICS

Further important contributions have been made by the Eastern Regional Research Laboratory toward the solution of problems connected with the conversion of lactic acid, which may be made by fermentation of milk sugar, molasses, corn sugar, and other carbohydrates, into acrylic acid esters. Two such esters, methyl acrylate and methyl methacrylate, are used commercially for preparing transparent resinous thermoplastics having important military and civilian uses, and from the results of experiments made at the Eastern Regional Laboratory it appears that other acrylates may also become important materials for the manufacture of resinous and rubberlike plastics.

The polymers of acrylic acid esters are of the "organic glass" type, and may be soft or hard, flexible or rigid, semisolid or solid, depending on the method of preparation. Some of them possess unusual stickiness, some are resilient and elastic like rubber, and some are hard and tough. The acrylic polymers and copolymers are of special interest because polymerization can be effected in mass, in water emulsion, or in organic solvents. Since different polymers having a wide diversity of properties can be made under different conditions, they have prospective uses as resins, plastics, rubber substitutes, impregnating and coating materials, and ordinary and pressure-sensitive adhesives.

In the commercial production of methyl acrylate, lactic acid is reacted with methanol to form the ester methyl lactate, which is then converted to the methyl-acetyl ester of lactic acid (methyl acetoxypropionate) by acetylation with acetic anhydride or ketene. The diester in vapor form is decomposed by heat (pyrolized) into methyl acrylate and acetic acid or other byproduct.

The research at the Eastern Regional Laboratory has been directed toward the discovery of short cuts, increase of yields, and improvement of processes and equipment for purifying the crude dilute lactic acid obtained by fermentation, for converting lactic acid into esters with methanol and other alcohols, for acetylating these esters into diesters with a minimum quantity of critical materials, and for pyrolyzing vapors of the diesters into methyl or other acrylate with a minimum of useless or less valuable byproducts. In some of the studies, laboratory experiments have been supplemented by experiments on a pilot-plant scale.

One result of this research has been the development of a new process that yields a substantial proportion of the desired diester

of lactic acid directly, together with methyl lactate, by treating lactic acid under suitable conditions with methyl acetate. The methyl lactate may be separated and converted into the diester by treating in the conventional way with acetic anhydride or ketene, but less of such costly and critical reagents is required for the total quantity of diester obtained. In a modification of the new process, the diester is made from lactic acid, acetic acid, and methyl alcohol.

Another result was the discovery that phenyl and certain substituted phenyl acrylates can be made in high yields by the pyrolysis of the corresponding phenyl-acetyl ester of lactic acid in a manner similar to the production of methyl acrylate by pyrolysis of methyl-acetyl ester of lactic acid.

The Eastern Regional Laboratory has also experimented on the preparation of ethyl and higher aliphatic esters of acrylic acid and the acrylates of carbocyclic and heterocyclic alcohols, in the expectation that some of them would be suitable for making new kinds of useful polymerization products. These new esters were made by the process of alcoholysis in which the methyl radical of methyl acrylate was displaced by the radical of another alcohol. Some of them seemed to have potential value for chemical industries.

Promising rubberlike plastics were prepared from esters of acrylic acid and other unsaturated chemical compounds derived entirely from agricultural products. Some of them had a tensile strength of around 1,800 pounds per square inch and a stretch (at breaking point) of 200 to 800 percent. These new rubber substitutes were named "Lactoprenes" to indicate derivation from lactic acid.

Continuous processes were developed for purifying crude dilute lactic acid obtained by fermentation, for making polylactic acids, and for making esters and diesters of lactic acid.

Eight scientific papers covering the work on lactic acid and on its derivatives and their pyrolytic decomposition products were prepared for presentation and publication. Some were prepared especially for a symposium on lactic acid and derived products to be held during the 1943 fall meeting of the American Chemical Society.

DETERIORATION OF FATS AND OILS REDUCED BY NEW PRACTICES

In connection with studies on the utilization of cottonseed and its products at the Southern Regional Research Laboratory, it was found that during storage of the seed a high moisture content favors the formation of free fatty acids by autocatalytic hydrolysis of the oil. When the moisture content was less than 10 percent there was no appreciable formation of free fatty acids during 6 months of storage, but when the moisture content was higher, fatty acids were formed rapidly.

Laboratory experiments with various treatments designed to prevent biochemical changes in cottonseed showed that treatment of moist seed with gaseous ammonia prevented heating in storage, decreased the hydrolysis of oil to free fatty acids and glycerin, and improved the color of the extracted oil in comparison with that of oil from untreated seed. A later experiment in which 1 ton of moist cottonseed was treated with ammonia gas before being put in storage showed that the laboratory method is applicable to large batches of seed. It was planned to apply the method to full mill-scale operation in the

fall of 1943. If found to be practical, general use of the ammonia treatment for moist cottonseed should follow. This would materially reduce the loss of oil from deterioration of cottonseed stored in moist condition.

Oil chemists have learned within recent years that edible vegetable oils naturally contain substances that retard oxidation and consequently the type of rancidity that is caused by oxidation. Unfortunately, the methods used for refining oils remove the greater part of the antioxidants, and highly refined oils are therefore more unstable than crude oils. Research at the Southern Regional Laboratory on the stability of cottonseed and peanut oils and of their fatlike hydrogenation products has increased the available knowledge regarding means for preventing rancidity both by control and preservation of the natural antioxidants and by the addition of synthetic antioxidants. Concentrates of the natural antioxidant alpha-tocopherol were prepared from peanut oil and its hydrogenation products. These were assayed for their content of antioxidant and their activity in stabilizing different fractions of peanut oil obtained in molecular distillation. The concentration of alpha-tocopherol or of antioxidant concentrate corresponding to maximum stability was determined for several vegetable oils and their hydrogenation products.

Several synthetic antioxidants were prepared and tested to determine their relative value for stabilizing fats and oils. Condensation products of catechol and aldehydes, and of naphthols and aldehydes, were found to have remarkable ability to inhibit oxidative rancidity, and to be equally useful for liquid and hydrogenated or hardened oils. Several derivatives of hydroquinone, especially the hexaldehyde and heptaldehyde derivatives, were also found to be extremely effective in stabilizing fats and oils against oxidative rancidity.

The Eastern Regional Research Laboratory has given much study to preventing rancidity in lard and other animal fats because of the relatively poor keeping qualities of such fats and the wartime necessity of preventing waste in edible fats and oils. In 1942 it reported that fat-solube compounds of ascorbic and isoascorbic acids acted as antioxidants when added to lard and gave promise of being practical agents for preventing rancidity. Considerable industrial interest was aroused by this report.

Further investigations on the use of fat-soluble compounds of ascorbic and isoascorbic acids as antioxidants for fats and oils showed that the effectiveness of ascorbyl esters depends chiefly upon their synergistic activity with natural oxidation inhibitors existing in or added to the fat, such as phospholipides and tocopherols. Especially good results were obtained when ascorbyl esters, soybean lecithin, and tocopherols were added to lard in the proportions of 0.03, 0.03, and 0.005 percent, respectively. With these concentrations of antioxidants, the stability of the lard was increased sixfold, and with twice these concentrations, the stability was increased tenfold, according to accelerated tests at high temperatures. This mixture of three antioxidants was found to be effective also for oleo oil, tallow, butter oil, oleomargarine, and seed oils. In fact, oil from corn, cottonseed, soybean, or wheat germ might be added directly to lard or other animal fat, in the proportion of 5 to 10 percent, to supply the tocopherol needed for the three-component antioxidant mixture.

Baking tests on crackers containing as shortening lard stabilized with the three antioxidants showed that the stabilizing effect survived the baking process. Likewise steam deodorization of lard containing the three antioxidants did not affect the stabilizing action of the antioxidant combination adversely. Steam deodorization of lard containing ascorbyl esters and soybean lecithin did cause some discoloration and removal of this discoloration with adsorbent material lowered the stability of the product appreciably. But even after deodorization and decolorization the lard had greater stability than the untreated lard as well as a white color and a bland taste.

At the Northern Regional Research Laboratory a chemical study was made of the formation and decomposition of the peroxides of unsaturated fat esters of soybean oil to obtain fundamental information needed for interpreting the role of these peroxides in the development of rancidity and other defects in edible oils. A clearer understanding of the chemical reactions involved in the development of rancidity would make it possible to modify the methods of processing and handling vegetable oils to make them more stable. The results of this study were prepared for publication.

SOLVENT EXTRACTION OF TUNG OIL MADE CONTINUOUS

The Agricultural Chemical Research Division has continued its efforts to promote the domestic tung oil industry, and especially to obtain the greatest possible quantity of oil from tung seeds grown in this country. Tung oil has important industrial uses and is a critical war material of which the Western Hemisphere produces very little; importations from China, which has supplied most of this country's needs in the past, cannot be resumed until the war with Japan is ended.

Previous work had shown that batch-type solvent extraction could be used for extracting commercial hydraulic press cake. The batch solvent process was also used to advantage with whole ground tung meal. However, certain inefficiencies inherent in the batch extraction process pointed to the desirability of using continuous solvent extraction.

Extensive pilot-plant experiments were made with a new type of continuous extractor which had given the better results; in this extractor the oil-containing material moves slowly through a long chamber of rectangular cross section, slightly inclined and divided into compartments having semicircular bottoms, over which the material is pushed by perforated revolving paddles against the solvent moving in the opposite direction.

After a number of tests on material prepared in various ways, and under various operating conditions, successful extractions were finally achieved with ground tung kernels and with fresh tung oil press cake, using normal hexane as the solvent and filtering the oil solution before evaporating off the solvent. Tung oil of good quality was obtained with an extraction efficiency of over 99 percent.

DOMESTIC SEED OILS MODIFIED TO FIND NEW USES

Further industrial tests have been made of the modified cottonseed oil which, as related in the last annual report of the Bureau of Agricultural Chemistry and Engineering, was developed by the Southern

Regional Research Laboratory as a substitute for palm oil in tin and terne plating. The tests showed that the modified oil is actually superior to palm oil for this use. While under normal conditions the cottonseed oil replacement would cost twice as much as palm oil, its useful life would be three times as long. The feasibility of replacing the entire present annual consumption of 40,000,000 pounds of imported palm oil with modified cottonseed oil was demonstrated.

A combination of controlled hydrogenation and low-temperature solvent fractionation was applied to the production of a hardened fat from cottonseed oil having the consistency and other properties of imported cocoa butter, extensively used in confectionery and pharmaceutical products. The new fat is, in most of its properties, very similar to cocoa butter, and other genuine plant-fat butters. Development of suitable commercial processing equipment should make possible the annual production of more than 50 million pounds of substitute fat which seems to be needed.

The combination of controlled hydrogenation and solvent fractionation was also applied to peanut oil to produce a replacement for olive oil in the textile industry. The product was tested commercially as a worsted spinning lubricant, and was reported to be superior to olive oil.

By chlorination of palmitic and other saturated fatty acids, followed by pyrolytic dehydrochlorination, the Western Regional Research Laboratory has produced unsaturated derivatives which yield alkali soaps similar to coconut oil soaps in water solubility, wetting and detergent power, and lathering qualities. Some of these derivatives have very high iodine values, which indicates that the process of chlorination and dehydrochlorination is adaptable to the production of highly unsaturated drying-oil acids from saturated fatty acids.

Numerous derivatives of long-chain fatty acids were prepared in an effort to find new organic plasticizers for certain of the major industrial resins, particularly the vinyl and cellulose ester types. For plasticizing vinyl resins, the fatty acid esters of glycidol proved particularly promising. The compounds in this group are high-boiling colorless oils or low-melting solids, insoluble in water and stable to heat and light. It was found that such compounds are unsuitable for plasticizing cellulose ester resins because of their long chain length and consequent incompatibility. Hence several ether esters of short-chain dibasic acids were prepared. Of these, ethoxyethyl fumarate was outstanding when used with cellulose esters.

Various compounds of fatty acids with the common amino acids were synthesized and tested for their ability to platicize proteins. Two of them, the *n*-lauroyl and *n*-palmitoyl derivatives of tyrosine, have been found to be compatible with, and good plasticizers for, casein and gluten. The added property of water-insolubility possessed by these compounds appears to be unique among plasticizers for proteins.

At the Northern Regional Laboratory progress was made in the development of methods for isolating technically pure linoleic acid, or a mixture of linoleic and linolenic acids, from soybean fatty acids and modifying this compound, or mixture, by a newly developed isomerization process into isomers of eleostearic acid for the preparation of esters resembling tung oil. Installation of pilot-plant equipment for liquid-liquid extraction and vacuum distillation frac-

tionation of soybean oil was completed near the close of the fiscal year and large-scale experiments were started. Many operation variables still remain to be studied.

COTTON PROCESSING RESEARCH DIRECTED TOWARD SPECIAL WARTIME USES

Research on mechanical processing of cotton at the Southern Regional Research Laboratory has been directed toward: (1) The preparation of lint cotton and cotton waste for chemical use in the manufacture of cellulose compounds with existing equipment designed for cotton linters; (2) the use of cotton, alone or in combination with other available fibrous materials, for manufacturing binder twine and other types of twine commonly made from imported hard fibers; and (3) the use of cotton for manufacturing a superior tire cord, especially adapted to tires made from synthetic rubber.

An experimental model of a disk-type cotton-cutting machine, especially designed for rapidly reducing lint cotton to short-length fibers resembling linters, was completed. Extensive tests proved that this type of cutter is much more efficient than any standard commercial type, as regards rate of cutting and cost of operation. Application was filed for a patent on this cutter and also for a patent covering a companion machine, a combined cotton cleaner and feeder of high capacity and radically new design. Specifications for the cotton cleaning and feeding machine were supplied to a manufacturer of smokeless powder to permit the construction of such a feeder for use in experimental work with cotton linters.

Under a special appropriation of $30,000 to finance the construction of a disk-type cotton cutter and accessory equipment in commercial sizes, detailed workshop and assembly drawings, lists of materials, and specifications were prepared, and the necessary priority certificate was obtained, but there was no response to the invitation for competitive bids. A contract was finally entered into with a machine manufacturing firm, and construction was expected to begin before the end of the fiscal year. Representatives of one of the United Nations requested complete plans and specifications for the disk-type cotton cutter, the use of which is contemplated in connection with a smokeless-powder plant. The supply of cotton linters for the manufacture of smokeless powder and for other chemical uses has been ample thus far, but there is no certainty that this will be true in future crop years. Construction and successful operation of the commercial-size, disk-type cutter will remove any danger of an insufficient supply of linter-type cellulose for use in smokeless powder, acetate rayon, and other products for war uses.

Field tests on three sizes of experimental binder twine, made entirely from cotton, showed that such twine had sufficient strength and could probably be used satisfactorily if a package could be developed to prevent collapse of the twine when the ball approached exhaustion. Experiments were then made on the construction of a stiffer cotton twine which would ball on the regular sisal-binder twine-ball winder. The results indicated that a usable cotton twine can be produced, but the cost is high compared with that of other possible substitutes for sisal twine. Other tests showed that twine made of hard fibers with cotton cores had satisfactory tying quality. This combination of cotton with hard fibers seems more practical than an all-cotton binder

twine, which is not required as long as other suitable materials are available. This fact is recognized by the Department of Agriculture, which recently initiated a program to subsidize the consumption of low-grade cotton as cotton-yarn cores for binder twine. Since the cotton content of such twines is about 30 percent, the program makes the limited quantities of available hard fiber go further.

A new cotton-utilization project, started by the Southern Regional Laboratory in November 1942 has for its objectives the improvement of cotton fiber and yarns for use in tire cords and a knowledge of how to prepare the best possible cotton tire-cord fabrics for use in making tire casings from synthetic rubber. Five outstanding varieties of cotton were selected, with advice from other units in the Department of Agriculture, as having fiber suitable for use in tire cords and as being produced in sufficient quantity to be of commercial interest. Two industrial firms cooperated in the production of experimental cord fabrics from these five varieties of cotton, one starting with the cotton itself and the other with yarns made by the laboratory. Also, one variety was made into experimental cord by the laboratory. This cord was subjected to a special treatment intended to improve its properties, after which it was woven into tire-cord fabric by an industrial firm.

All of the experimental tire-cord fabrics were delivered to a tire-manufacturing firm and were evaluated comparatively by its usual laboratory tests. On the basis of the results, the fabrics made from three lots of yarn spun at the Southern Regional Laboratory, and representing three out of the five varieties of cotton, were selected for use in manufacturing an initial lot of S-4 test tires, 90 percent of the rubber being synthetic and 10 percent being natural.

Up to the end of the fiscal year, only indoor wheel tests had been completed on tires representing one variety of cotton.

CHEMICAL PROCESSING MAKES COTTON MORE USEFUL

In its research on adapting cotton to new uses and making it more serviceable for established uses, the Southern Regional Research Laboratory employs not only mechanical processing but also chemical treatments to modify the composition or the physical properties of the fibers, or both, and chemically prepared finishing materials for imparting desirable properties to cotton yarns and fabrics. In some cases, research on mechanical processing and research on chemical modification or additive finishing of cotton yarns and fabrics both contribute toward the accomplishment of the objective.

In connection with the research to develop improved types of cotton tire cord, a machine for treating, stretching, and drying cords on a semiplant scale and a similar smaller machine for laboratory tests were designed and built. These were used to apply mercerizing and other chemical treatments to experimental cords. Another machine was built and used for continuously acetylating, washing, and drying yarns before twisting them into cords.

Mercerization, treatment with amines, and partial acetylation all reduced the loss in tensile strength of cotton cords when tested at high temperature (130° C.). Cords that were mercerized and treated with a cation-active wetting agent to increase adhesion between cord fabric and rubber stock showed the least loss of tensile strength due to heat.

Mercerization of yarns not under tension increased the flexing durability of cords prepared from them. Treatment of plied yarns with a cationic finishing agent increased flexing durability more than threefold in the case of cords made from yarns mercerized under tension, and more than five-fold in the case of yarns mercerized without tension. Flexing strength, as determined at 121° C., was also improved by treatment with ethylamine and heptylamine.

With the cooperation of several other Government agencies, experiments were made with rotproofing treatments for osnaburg, a coarse cotton fabric being used to supplement short supplies of jute burlap, to make it last longer when used in sandbags for military and civilian defense purposes. Treatment with furfuryl alcohol resin in moderate concentrations was found satisfactory, when the osnaburg was subjected to soil burial tests. A detailed study was made of the value of cuprammonium compounds and cuprammonium-fluoride mixtures, when used in various concentrations as rotproofing agents for sandbag fabrics. Progress was made in developing an improved method of acetylating cotton thread and fabric to impart high resistance to rotting.

In research on permanent fireproofing of cotton fabrics compounds of organic bases with amidophosphoric acid, synthesized in the laboratory, were found to impart satisfactory flame resistance, but the treatments were not sufficiently stable to washing. A resinous organic derivative of ammonium sulfamate, which was developed as a fireproofing material, showed considerable resistance to removal when the fabric was washed with cold water.

In an attempt to improve the current commercial finish for fire-, water-, and weather-resistant cotton duck used to meet army specifications, and to develop a finish for lightweight fabrics that would be permeable to air as well as fast to washing, changes were made in the regular procedure both as to materials used and the manner of compounding the ingredients. Encouraging results were obtained and the process is being developed intensively. The finish appeared to be unaffected by repeated laundering.

By means of a modified mercerization process applied to loosely woven cotton fabric of cheesecloth type, the Southern Regional Laboratory has produced an improved cotton bandage having new and desirable texture and elastic properties that make it stay in place and conform more readily than the ordinary gauze bandages to the contour of extremities. The elastic properties result from shrinkage of the yarns, which may be brought about so as to affect the bandage lengthwise, crosswise, or in both directions. Shrinkage of 20 to 40 percent both lengthwise and crosswise gives a fabric that can be easily stretched in any direction and that tends to recover its original dimensions when released from tension. The yarns of the bandage are kinked by the treatment and the general effect is a roughened surface in the fabric which imparts valuable nonslip properties. The limited clinical trials that have been made thus far with experimentally treated bandages were reported as being very successful. There will be some manufacturing problems in applying the treatment commercially, because, in contrast to the usual textile processing, the bandage fabric must be handled without tension after shrinkage has

taken place. It is believed, however, that the new bandage material can be manufactured without prohibitive changes in the equipment of plants now producing surgical gauze.

The results of laboratory tests on the effects of various reagents on the properties of chemically modified cotton led to an investigation that concerns the preparation of cellulose nitrate for use in smokeless powder and in cellulose plastics. It was found that nitrated cellulose, made from purified cotton linters or lint cotton cut to short lengths, can be stabilized quickly and economically by washing several times with cold water containing a small quantity of ammonia. As judged from the laboratory experiments, this process seems promising because, in comparison with present commercial practice, it would eliminate the use of heat for boiling the numerous lots of wash water, would shorten the time of treatment to a fraction of that now required, and would yield a product of improved stability.

NEW USES IN PROSPECT FOR AGRICULTURAL PROTEINS

In its research on the industrial utilization of agricultural products and byproducts, the Bureau of Agricultural and Industrial Chemistry is confronted with many problems relating to the protein constituents of agricultural products, especially when the proteins are unsuitable or not disposable for use as food or feed. For example, in the recovery of starch from damaged wheat, or in certain fermentation processes, the protein byproduct may be unsuitable for feed and so nonfood uses are sought. Other examples of inedible protein materials are the oil press cakes from tung seeds and castor beans and the keratin-containing materials—hair, hoofs, horns, and feathers. Although such proteins can be used for fertilizers, other more profitable uses are desirable.

At the Western Regional Research Laboratory means have been devised for dispersing the keratins of feathers and other animal wastes into liquids so they can be used as adhesives. Glues prepared from poultry feathers were found from laboratory tests to be suitable for various uses. For example, fiber containers coated on the inside with a diluted dispersion of the feather keratin were impervious to paints, paint oils, and edible oils, and could serve as satisfactory substitutes for metal containers. Preliminary experiments have indicated that dispersions of keratin in dilute alkaline hydroxylamine solutions are better than those in alkaline sulfide solutions for use as glues. It is believed that keratin glues for plywood can be developed which will equal in performance the glue made from casein or soybean meal, thus releasing these edible proteins for use in foods to help meet wartime emergency requirements.

Artificial fibers have been prepared from dispersions of such proteinaceous materials as feather keratin, egg albumen, and wheat gluten in nearly neutral solutions containing a commercial wetting agent or detergent of the alkyl-aryl sulfonate type by extruding the viscous liquid through a spinnerette into a coagulating salt solution. The filaments, consisting of protein-detergent complex, are treated with a mixture of acetone and water, which removes the detergent and regenerates the protein. They are then stretched 200 to 400 percent while being subjected to steam at atmospheric pressure. This treatment is intended to arrange the chainlike molecules

parallel to each other and thus strengthen the filaments. After stretching, the filaments can be made more water-resistant by treating with a tanning agent, such as formaldehyde. The tensile strength of the dried fibers is higher than that of wool and approaches that of silk of the same diameter, but the wet strength is deficient and needs to be improved for certain uses.

A thermosetting plastic molding composition, in which nearly a third of the phenolic resin commonly used is replaced with a slightly higher proportion of keratin from feathers or other source (using proportionately less wood flour), was developed at the Western Regional Laboratory. The composition can be molded at the same temperatures and with the same operating cycles as are used for phenol-formaldehyde molding compositions and can be colored more satisfactorily with dyes. However, the flexural strength of the molded articles is about 10 percent less.

At the Northern Regional Laboratory a notable improvement was made in the color and taste of soybean protein and soybean meal by extracting the oil from flaked soybean kernels with alcohol under controlled moisture conditions. The results indicated that completion of this investigation will expand the industrial consumption of soybean protein for fibers, plastics, and paper coatings and of soybean flour for cheese and milk substitutes.

The soybean meal-phenolic resin molding powders previously developed by the Bureau have been improved with regard to flow properties without sacrificing water resistance by substituting a new method of preparing the soybean meal for the earlier one of treating it with formaldehyde. The new method consists of leaching the soybean meal with acidified water (pH 4.0 to 4.6) to remove water-soluble materials, followed by heating to change the solubility properties of the protein. Soybean meal treated in this way may be used to replace part of the phenolic resin and wood flour in the usual molding-powder formula. Replacement of wood flour by soybean meal causes a rapid increase in flow, whereas addition of increasing amounts of soybean meal to phenolic resin and wood flour, mixed in constant ratio, results in a gradual decrease in flow. These two effects can be balanced to obtain the desired flow properties with a lower quantity of phenolic resin. The wet-mix method previously described was found to be better than the dry-mix method for incorporating the soybean meal with phenolic resin and wood flour in that it reduces to some extent the loss of impact strength which results from substitution of phenolic resin with soybean meal.

At the Northern Regional Laboratory, zein, the alcohol-soluble protein of corn, was adapted to commercial use as a liquid coating and plastic material, replacing shellac for some purposes, by showing how to keep its solution from gelling and how to increase the elasticity, strength, and stability of its films by incorporating suitable plasticizers. Plasticizers for zein were found in hydrogenated and unhydrogenated dimerized fat acids prepared from soybean oil. A mixture of dimerized fat acids (left after distillation of monomeric acids from alkali-isomerized soybean fatty acids), which was developed as an intermediate for the manufacture of the rubber substitute Norepol and similar products, is now available commercially. The principal constituent of this mixture, dimeric linoleic acid, was found to be responsible for its plasticizing action but it is not practical to iso-

late this compound for industrial uses under existing economic conditions. The residual dimerized fat acid mixture is suitable for plasticizing zein coatings on food-packaging materials because it has no odor or taste, and imparts water resistance, resistance to penetration by moisture vapor, and resistance to penetration by vegetable oils. In laboratory trials by a bottling company, zein films stabilized against water absorption and plasticized by methods developed by this Bureau were found satisfactory for coating the resilient disks used for lining bottle caps. Plasticized zein films have also been made which resist penetration by turpentine.

Zein was found to be adapted for use in conjunction with commercial paper-coating materials; laboratory tests showed that it is perfectly compatible with the urea-formaldehyde and melamine-formaldehyde resins so widely used in the coated-paper trade.

At the Southern Regional Laboratory it was found that when peanut kernels are stripped of their red skins by blanching before they are ground and pressed for oil, and when the white-skinned unblanched kernels of certain varieties are used, the protein obtained by extracting the oil press cake with dispersing agents and precipitating is equal or even superior to available commercial soybean protein as regards color. The utilization of peanut protein for adhesives, water paints, paper coatings and artificial fibers has been hindered heretofore by its objectionable color.

Plywood glues that were considerably better than the minimum requirements in Army and Navy specifications for water-resistant casein glue were prepared from peanut meal and from the protein fractions separated from peanut meal.

At the Eastern Regional Laboratory artificial fibers were made experimentally by extruding a heated plastic mixture of casein and water into air, thus obviating the need for a precipitating bath as employed in the present manufacture of artificial fiber. Coarse filaments (0.008 to 0.016 inch in diameter), apparently suitable for use as substitutes for horsehair and hog bristles, were made by employing this process and subjecting the resulting filament to physical and chemical treatments to improve the tensile strength, flexibility, and water resistance. Good-looking brushes for various purposes were made successfully from different lots of such filaments having appropriate diameters and flexibilities. Large sample lots of the artificial bristles were supplied to manufacturers for making experimental brushes. Paint brushes as well as various types of dry-use brushes are under consideration.

Casein freshly precipitated from skim milk could be used directly in such an extrusion process; therefore the cost of drying and grinding could be avoided if the fibers were made at the casein-producing plant.

Results of research on the utilization of wheat protein are reported under Wheat Becomes Important to Chemical Industries.

USEFUL CHEMICALS DERIVED FROM TOBACCO

In connection with studies of new nicotine compounds for use as insecticides, several additional nicotine-metal double salts were prepared. Of the many double-salt nicotine compounds which have been prepared by the Eastern Regional Research Laboratory and subjected to preliminary testing by the Bureau of Entomology and Plant Quarantine against chewing insects, some five or six appear to have

notable toxicity. These include copper and zinc combinations which may be suited to commercial production.

A series of new nicotine-metal compounds of the nicotinamino type was prepared by reaction of the free alkaloid with metal salts. Several of them showed unexpected stability and desirable physical properties, and in addition, their preparation was simpler than that of the double salts just mentioned. Tests of their insecticidal action were begun.

Further progress was made in the development of improved methods for the oxidation of nicotine to the antipellagra vitamin, niacin (nicotinic acid). In tests with various catalysts and combinations of catalysts, yields from the liquid-phase oxidation process were increased to 75 percent of theory. It was demonstrated that the method could be used to prepare nicotinic acid not only from nicotine but also from the coal-tar distillation products, beta-picoline and quinoline.

Studies of the catalytic vapor-phase oxidation of nicotine also were continued. This process results in the formation of a partial-oxidation intermediate, nicotinonitrile, in yields slightly greater than 50 percent of theoretical. This intermediate can be converted by hydrolysis into nicotinic acid or into nicotinamide, which also has pellagra-preventive properties. New methods were developed for the conversion of nicotinonitrile to nicotinamide and for the solvent extraction of the latter from the reaction mixture.

Studies were made of the economic extraction of citric and malic acids from tobacco residues left after removal of nicotine. The most efficient method involved pretreatment of the residues with sulfuric acid, followed by water extraction and recovery of crude calcium citrate and malate. These salts were prepared on a fairly large scale for a study of methods of recovering the purified acids.

Rutin, a flavone glucoside, was isolated from tobacco in pure form. Since it was believed that rutin might have vitamin P activity, arrangements were made for testing it under clinical conditions. It was indicated from a 6-month trial that rutin has merit in the treatment of disease conditions associated with capillary fragility. Cooperative tests by the Bureau of Animal Industry showed that in long-continued feeding rutin was harmless to the laboratory animals used.

TURPENTINE AND ROSIN YIELD NEW USEFUL DERIVATIVES

Much has been learned about the chemical components of the very complex substances turpentine and rosin, and of the pine "gums" from which they are derived, through years of fundamental research by the Naval Stores Research Division. Means for separating pine oleoresin and turpentine components have been devised and derivatives of individual components have been prepared and studied with regard to commercial possibilities. New resin-acid products and improved types of rosin and rosin products have been developed for industrial uses.

The report for the fiscal year 1942 mentioned two unsaturated chemical compounds, allo-ocimene and myrcene, which had been obtained by the pyrolytic decomposition of alpha-pinene and beta-

pinene, respectively, the principal components of turpentine, and their potential value for promoting drying of semidrying oils and for making synthetic resins, rubber, and other products. It also stated that efforts were being made to produce esters of rosin and glycols that could be substituted for rosin glyceride in varnishes, thus releasing glycerine for war uses.

In the early part of the fiscal year substantial quantities of myrcene were made in the laboratory from beta-pinene that had been isolated from turpentine, and samples were supplied to several commercial firms for additional experiments to determine the value of this material for making synthetic rubber. Reports on the results of these experiments were, for the most part, encouraging. They indicated that good synthetic rubbers, in both solid and latex form, can be made from myrcene alone or by copolymerizing myrcene with one or more of the intermediates being used commercially in the production of synthetic rubbers.

Myrcene is now produced on a small scale from the beta-pinene of turpentine by the process developed by the Naval Stores Research Division, even though the yield by this process amounts to only about 20 percent of the turpentine represented or about 60 percent of the beta-pinene actually used. One firm is already using myrcene in manufacturing synthetic rubber on a small scale, especially for adhesive use, by copolymerizing it with styrene alone or with styrene and isoprene.

Experiments in cooperation with Government and industrial laboratories are still in progress to determine if myrcene polymers or copolymers can be used to increase the adhesive properties of Buna rubbers or to replace the natural rubber used to insure adherence of the layers comprising the carcass of synthetic-rubber tires.

Preliminary experiments were made in an effort to develop an efficient pyrolytic method for preparing the synthetic-rubber intermediate isoprene from gum spirits of turpentine. Various turpentine constituents and derivatives, including allo-ocimene, alpha-pinene, beta-pinene, myrcene, and dipentene, were pyrolyzed, using two methods. Dipentene gave by far the best yields.

Samples of allo-ocimene prepared in the laboratory and samples obtained from a commercial source were sent to the research laboratories of several industrial firms for cooperative experiments on utilization. Confidence was expressed by one firm in the prospects for using allo-ocimene for producing synthetic resins.

A varnish thinned with a mixture of mineral spirits and allo-ocimene (80:20) set to the touch in 4 hours, whereas the same varnish thinned with an equal quantity of mineral spirits alone required 6 hours for drying to the same degree. However, this advantage of faster drying seems to be offset by a disadvantage because, on standing in the containers, the varnish containing allo-ocimene formed a thick skin, whereas the other did not.

Viscous liquid polymerization products of rosin having drying or film-forming properties were prepared by heating various kinds of rosin and modified rosins to fairly high temperatures in contact with mineral acid or other catalyst. Some of these "rosin drying oils" showed promise of finding use in protective coatings and in products for waterproofing paper containers. When tall oil, a liquid byprod-

uct of paper-pulp manufacture from coniferous wood which is composed of resin acids and fatty acids in about equal quantities, was subjected to a similar treatment, the resin acids were converted into a complex mixture of hydrocarbons resembling rosin oil. This product could be separated from the unchanged fatty acids by treating with alkali solution to form soaps of the latter and extracting the mixture with organic solvent.

Some of the turpentine derivatives and rosin compounds prepared in the laboratory were tested to determine their value for use in concrete-curing paints, which are replacing wet burlap for preventing too rapid drying of freshly laid concrete pavements and roads. Each of the materials was dissolved in or diluted with an equal volume of volatile mineral spirits, and the preparations were submitted to the Highway Testing Laboratory of the Bureau of Public Roads for comparison with one of the best of the commercial products used in curing concrete roads. The final report was not received before the end of the fiscal year, but a preliminary report indicated that the preparation made with zinc rosinate was as effective as the commercial product. After zinc rosinate, the report listed rosin drying oil having a high acid number, limed rosin, untreated rosin, and other products in descending order of effectiveness. The turpentine derivatives, allo-ocimene and unrefined alpha-pinene pyrolysate, were not suitable for use as such because when spread in a thin film the evaporation was too rapid to permit the degree of oxidation or polymerization required to form an impervious film. Allo-ocimene and pyrolysates of alpha-pinene rich in allo-ocimene were oxidized and the oxidation products were prepared for supplementary tests to determine if they are suitable for use in concrete-curing paints.

In experiments on the value of rosin-lactic acid reaction products as substitutes for rosin glyceride in varnish making, it was found that the product obtained when using 2 mols of lactic acid per mol of rosin was quite dark in color, but yielded a varnish having better water resistance than the standard spar varnish. This new varnish was not suitable for use with basic pigments like zinc oxide because the resin, which had a high acid number, reacted with such pigments, causing livering. Neutralization of the acid with lime or zinc oxide after the completion of the reaction between the rosin and lactic acid destroyed the water resistance of the varnish made from the product.

NEW FOOD PRODUCTS MADE FROM FRUITS

In Florida as many as a million boxes of tangerines have been wasted annually because of size and grade restrictions. It was demonstrated by the Agricultural Chemical Research Division that it is possible to combine the recovery of flavoring oil and cattle feed with the manufacture of two types of vacuum-concentrated byproducts which have high food value as well as excellent flavor and keeping quality. Bland sirup was made from tangerine juice with the retention of 75 percent of the original vitamin C content. This sirup can be stored at tropical temperatures without deterioration, which makes it suitable for use where citrus juices or concentrates cannot be easily provided in good condition. A tangerine beverage-base concentrate can be used like other citrus concentrates in carbonated

beverages and food products where the fruit flavor, minerals, and vitamin C content make it desirable.

A study was made of vitamin C losses during processing of citrus juice in 16 commercial plants in order to determine what methods were most effective in preserving quality. Technical assistance was given to two commercial plants which installed vacuum-concentrating equipment for handling citrus juice. Aid was also given to commercial ventures into the packing of clear and cloudy lime juice and other lime products.

Several methods were demonstrated for reducing the moisture content of marmalade stock as a means of reducing the shipping facilities required for handling the product. One method consists of packing the sliced peel with sulfur dioxide in barrels, concentrating the juice to 65 percent soluble solids, and preserving it separately with sulfur dioxide in barrels. When the peel and concentrate are combined with sugar and water and boiled in manufacturing marmalade, the sulfur dioxide is substantially removed, and the resulting marmalade is considered equal in quality to that made from fresh marmalade stock.

A process was developed for preparing a pectin concentrate from grapefruit pomace. This involves leaching the pomace with water to remove flavoring matter and other undesirable constituents, followed by pressing, drying, and grinding. Treatment for pectin recovery consists of heating the dried pomace with acidulated water under controlled conditions and removing the insoluble cellulosic material by straining through cloth. Specifications for the manufacture and testing of the dried pomace were prepared and submitted to the Agricultural Marketing Administration for use in purchasing the product for lend-lease shipment to Great Britain.

A method was discovered for the preparation of powdered orange juice without the use of added spreading material, and a public-service patent was applied for.

Methods were developed for preparing a variety of products from papayas and for dehydrating this fruit. The papaya contains generous quantities of vitamin C and cryptoxanthin—one of the precursors of vitamin A—and the dehydrated material has a bland, pleasant flavor resembling candy, which makes it suitable for use in concentrated rations without modification. The fruit and pulp can be combined with orange and grapefruit juice in preparing canned foods of high vitamin content, attractive flavor, and satisfactory storage properties.

BLAND APPLE SIRUP FINDS NEW USES

A bland sirup developed from low-grade apples by the Eastern Regional Research Laboratory has found wide acceptance as a humectant to replace glycerin in conditioning cigarette tobacco. Rapid progress from the pilot plant to the commercial stage resulted in the production by five processors of more than 3,000,000 pounds of sirup from the 1942 apple crop. Since the use of glycerin in tobacco was entirely discontinued in March of 1943, it is estimated that the tobacco industry could absorb 30,000,000 pounds of apple sirup from the 1943 crop, if fruit and facilities are available for its manufacture.

During the year assistance was given to sirup processors in expanding their capacity and in the elimination of certain difficulties encountered in commercial-scale operations. Also, attention was given to the modification of the sirup for uses other than as a tobacco humectant. Especially promising outlets are pharmaceutical sirups, dentifrices, and fruit ices. Some of these uses indicate an advantage for apple sirup that will assure its peacetime production.

PECTIN FROM FRUIT WASTES ADAPTED TO IMPORTANT NEW USES

The Western Regional Research Laboratory has given much attention to the improvement of methods for extracting pectin from fruit wastes, such as peels, cores, and press residues from fruit-processing plants, and to the purification or modification of pectin for special food, medicinal, bacteriological, and industrial uses.

A new method was developed for extracting pectin from citrus fruit peels and apple pomace. This is based on the use of a slightly acid solution containing sodium hexametaphosphate, which forms soluble complexes with the calcium and magnesium compounds in the fruit material. This method, which seems adaptable to use in continuous processing, yields about 20 percent more pectin from apple pomace, and about 40 percent more from citrus peel, in shorter periods of extraction and with lower proportions of water, than can be obtained from the same materials by the techniques commonly used.

In response to many requests from manufacturers of fruit jellies and preserves, a procedure was developed for preparing a liquid concentrate of pectin from apple pomace with equipment ordinarily available in fruit-preserving plants.

Since the isolation of pectin itself from pectin extracts by the usual method of alcohol precipitation is costly, the requirements for obtaining pectin of good quality by spray or drum drying of the extracts were studied. Among such requirements are a reduction in the content of minerals naturally present in pectin extracts and also removal of lead and arsenic spray contaminants when apple pomace is used as the source of pectin. From experiments on a laboratory scale, it appears possible to accomplish these purposes by filtering the pectin extracts through materials having ion-exchange properties. Three commercial cation-exchange materials have been tried for removal of lead and reduction in calcium and magnesium content, and three commercial anion-exchange materials for the removal of arsenic, to determine their effectiveness and relative capacities. The most efficient ion exchangers are to be used in further experiments on a larger scale to determine if their use for this purpose is practical.

An investigation on the suitability of pectin for replacing tragacanth or other water-soluble gums in the preparation of medicated jellies and ointments of the emulsion type for treating burns and minor surface injuries and infections was completed. From studies of the compatibility of various medicaments with the supporting media and the stability of the products, it was found that pectin serves very well as a substitute for the imported gums formerly used.

Tests in bacteriological laboratories have shown the suitability of a special pectin compound proposed for use as a substitute for agar in preparing nutrient media for culturing the micro-organisms. This compound is a mixed salt of "fibrous" pectic acid and may be pre-

pared from commercially available fibrous sodium pectate by adding a small proportion of soluble calcium salt. Not only is there a satisfactory substitute for agar available when needed, but also the supply of agar in this country can be made to last longer by reclaiming used agar for further bacteriological use by a simple method devised in the Western Regional Laboratory.

The Eastern Regional Laboratory has carried out further experimental work in cooperation with the Delaware Agricultural Experiment Station on the development of methods for preparing from apple pomace demethylated pectin derivatives called low-sugar jellying pectinates. With these products jellies containing 35 percent or less sugar can be prepared, whereas the usual sugar content of jellies is around 65 percent. Various improvements were made in the acid-demethylation method and also in the enzyme-demethylation method. The acid-demethylated pectinates were found to be better for making low-sugar jellies.

It was found that the extraction of pectin from apple pomace with oxalic acid, instead of dilute mineral acid, gave higher yields at lower temperature, and that the milder conditions of acidity and temperature improved the quality of the product. Of course, the oxalic acid must be entirely removed because it is poisonous. Experiments showed that precipitation of pectinates by alcohol is better than precipitation with aluminum salts, especially in the preparation of enzyme-demethylated pectinates, because any added polyvalent ions are difficult to remove and, if not removed, interfere with dispersion and reduce the gel strength of the product.

FRUIT AND VEGETABLE WASTES POTENTIAL SOURCES OF IMPORTANT PRODUCTS

At the Western Regional Research Laboratory *Torula* yeasts were grown on juice from culls and wastes of six fruits in yields equal to those obtained commercially with *Torula* grown on wood-sugar hydrolysates, and superior to commercial yields of *Saccharomyces* (baker's yeast) grown on a molasses substrate. The yeasts appear to be useful as a source of proteins for animal feeds.

To provide an adequate basis for commercial operations, a study was made of various factors influencing the suitability of asparagus-butt press juice for the culture of *Bacillus brevis*, an organism which produces the germicide, tyrothricin. Increased yields were obtained by the addition of suitable nutritional supplements to the juice. The progress of this work was greatly accelerated by the development of a new physico-chemical test for the tyrothricin content of culture liquids. The method of isolating tyrothricin from solution was improved.

Continued progress was made in the use of synthetic resin ion exchangers for recovery of tartaric acid from grape pomace and distillation residue rejected as wastes in making wine and brandy. The process involves the passage of dilute tartar-bearing liquids, such as still slop or pomace extract, through a filter bed of cation-exchange resin to remove metallic ions and then through one of anion-exchange resin to fix the acid. From the latter the acid is removed by soda-ash solution, with concurrent regeneration of the exchanger for the next cycle, and precipitated as calcium tartrate of excellent purity.

It was determined that the anion-exchange resins thus far examined can be expected to fix, from a normal still slop containing 0.2 to 0.3 percent of tartaric acid, about 5 pounds per cubic foot per cycle for a life period estimated at 100 cycles. The performance appears sufficient to render the operation commercially profitable at the present high price of the crude material.

In cooperation with a California vineyard association, a pilot plant for recovery of tartaric acid by means of ion exchangers was designed, built, and successfully operated during the last season.

Small-scale pilot trials by the Southern Regional Laboratory indicated the practical possibilities of a simple diffusion procedure for removal and recovery of soluble sugars and protein from sweetpotatoes prior to starch extraction. The sugars were shown to support rapid growth of feed yeasts.

Other investigations showed that high-grade pectin, comparable in quality to citrus pectin, can be readily extracted from the residual pulp, obtained as a byproduct of sweetpotato-starch manufacture, in yields as high as 40 pounds per ton of sweetpotatoes processed.

A comprehensive survey was made by the Eastern Regional Laboratory of large-scale vegetable wastes, such as pea vines, which showed that many of them have high nutritive value because of their content of protein, carotene, and riboflavin, and when dried should be a valuable supplement to the supply of animal feeds. Such wastes were successfully dehydrated without significant loss of nutritive properties. A method was devised for separating stems and midribs from the more valuable leafy portions of the wastes.

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